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# Synthesis and Properties of Carbonized Silicate Ceramics

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## Abstract

Temperature treatment of compositions based on red clay, grinded wastes of basalt (tezontle) and cullet makes it possible to reduce the sintering temperature and sintering time of ceramic products of different applications within the framework of a traditional technology, i.e., realize an energy-saving technology. The principle of formation of low-temperature eutectics is the basis for the energy-saving technology. The use of biowaste of sewage water treatment plants (WAS) enables us to perform plastic formation of products without addition of free water, thereby realizing a water-saving technology. The sintering of mixtures under conditions of a shortage of oxygen (in argon) leads to the formation of carbonized porous ceramics. Depending on the sintering regime, ceramics of different application (from building ceramics to adsorbing ceramics) can be obtained.

**Keywords:** Red Clay; Glass; Tezontle; Biowaste; Argon; Sintering; Ceramics.

## 1. Introduction

It is known that for the manufacture of porous ceramics, carbon-containing additives of different nature are often used [1–10]. Depending on the content of burnout additives and the sintering regime, different values of the porosity and mechanical properties of bricks made of red clay can be obtained [11–17]. Treatment in a redox atmosphere, in turn, is accompanied by both the incomplete burnout of formed carbon and formation of CO, which promotes the transformation the part of $Fe_2O_3$, which is present in red clays, into FeO, and to formation of $Fe_3O_4$. It was established that the presence of iron protoxide leads to an increase in the amount of melt and, as a result, to a decrease in the temperature of solid-state reactions, which lead to the formation of different spinels [16–20], and, therefore, to a decrease in the sintering temperature and time of brick products [19–22]. In view of the fact that, at a large content of a carbon-containing additive in the initial clay and in an accelerated sintering regime at deficit of oxygen, free carbon can be present in the synthesized ceramics [23–26], we suggested that this type of ceramic products can obtained a number of new physicochemical properties and consequently, to find new engineering and technical applications.

The objective of the present work is the development of energy- and water saving synthesis of carbonized porous silicate ceramics based on red clay with additives of industrial wastes and investigates properties of the obtained ceramic material depending on the sintering regime. Note that, in recent years, the recycling of the large-scale anthropogenic wastes in production of new types of ceramics (bricks), are extensively investigating [27–33]. This is why the aim of the present work is to seek a technologically simple solution on the processing of wastes of the stone-working industry, cullet, and waste of active sludge (WAS) from sewage water treatment plants, as this is ecologically important problem.

## 2. Experimental Technique

In the present work, several types of bricks obtained from red clay and three powder mixtures, namely, red clay–milled glass, red clay–tezontle, and red clay–milled glass–tezontle, were investigated. As a plasticizer, water-containing wastes of active sludge were used (40 and 60 g). The content of clay was varied from 100 to 25 wt. %, the content of glass was varied from 60 to 25 wt. %, and the content of tezontle was varied from 50 up to 25 wt. %. The size of glass and tezontle particles was 60 μm ≤ $d$ ≤ 250 μm. Laboratory briquettes of dimensions 120 × 60 × 20 mm were plastically formed and burnt in the temperature range 800–1000 °C for 1, 4, 8, 9, and 12 h. The burning of specimens was performed under conditions of a shortage of oxygen. The procedure was as follows. The formed specimens were put into a vacuum chamber. The chamber was evacuated to a pressure of $10^{-2}$ mm Hg and then blown by argon for 10 min. Thereafter compacts were put into tightly sealed containers inside the chamber, and then containers with specimens were subjected to temperature treatment in a muffle kiln.

The synthesized products were investigated by X-ray diffraction (XRD) in Cu $K_\alpha$ radiation (D2 PhaserBrukerdiffractometer). The contents of oxides and carbon in specimens were determined with use of an X-ray Fluorescence S8 TIGER spectrometer (Bruker). SEM and EDS measurements were performed with a LEO-1450 VP scanning electron microscope, HITACHI SU5000 field emission scanning electron microscope (FESEM). Investigations of water absorption by specimens, compression tests, and fracture tests were carried out according to standard techniques. To assess the adsorbing properties of specimens, aqueous solutions of methylene blue (MB) were used. Adsorption properties were studied by the UV-Vis method with the use of an USB4000-XR1 Ocean Optics spectrometer and calibration curves.

## 3. Results and Discussion

### 3.1 Initial materials

Figure 1 shows X-ray diffraction patterns of the initial components of the used mixtures, from which it follows that, along with cristobalite, red clay contains quartz and feldspar. Tezontle contains a number of aluminum silicates typical of basalts (chain and frame basalts). Used milled cullet is amorphous. WAS consist of an amorphous phase (organic matter) and inorganic components (clay mineral, silica, and feldspars), which enter sewage water treatment plants together with sewage water. It is seen in Fig. 2 a that the WAS sludge is similar to slime. Only after temperature treatment, inorganic solid particle are clearly seen (Fig. 2 b). It is evident from Table 1 that, along with the dominant carbon component, WAS contains a number of oxides. Note that the composition of WAS correlates with the composition of a series of compositions used for the synthesis of wall ceramics (bricks) and low-melting glasses [16, 17, 25, 26, 33]. This is why their use must not lead to a substantial change in the phase composition of the ceramics.

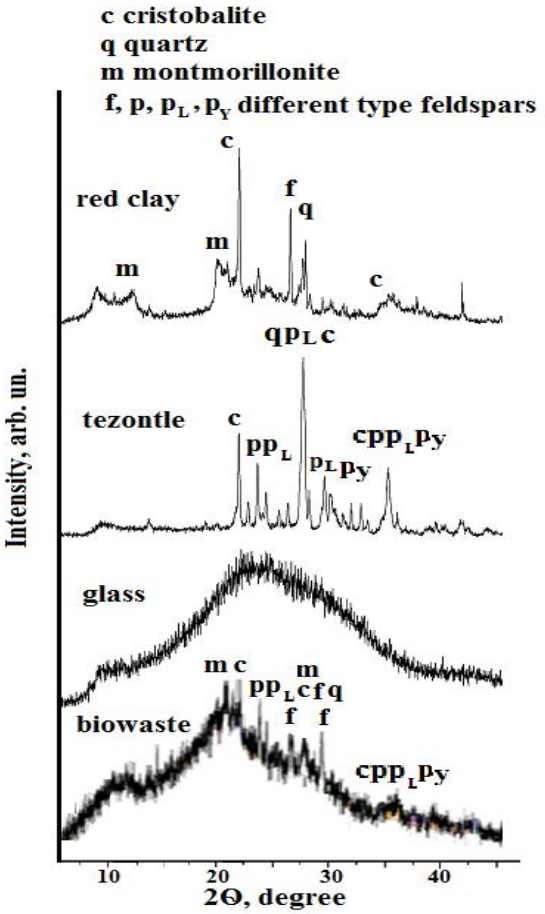


**Fig 1: Fragments of X-ray diffraction patterns of the initial components of mixtures.**

### 3.2 Ceramics

After temperature treatment of binary and ternary mixtures consisting of red clay, milled low-melting glass, and tezontle with the use of WAS and at a deficit of oxygen, an intensive halo is registered, which indicates the presence of carbon in the specimens (Fig. 3). Note that, after sintering of these mixtures in air, this intensive halo is absent due to the burnout of carbon in the ceramics (Fig. 3 a, c, d, see the lower parts of the X-ray diffraction patterns). This means that products sintered in an argon atmosphere have physicochemical properties differing radically from those of products sintered in air.

**Table 1. The composition of the WAS before and after carbonization in argon (the 7g of material).**

| Initial WAS | | $T_{tr.}$= (600-800)°C, $t_{tr.}$= (30-120) min | |
|---|---|---|---|
| Formula | Content,% | Formula | Content,% |
| C | 99.10 | C | 66.90 |
| $SiO_2$ | 0.26 | $SiO_2$ | 20.37 |
| CaO | 0.12 | $Na_2O$ | 3.51 |
| $Al_2O_3$ | 0.1 | CaO | 3.07 |
| $Fe_2O_3$ | 0.11 | $Al_2O_3$ | 1.41 |
| $SO_3$ | 0.17 | $Fe_2O_3$ | 1.40 |
| $P_2O_5$ | 0.06 | $SO_3$ | 1.15 |
| CuO | 0.04 | $P_2O_5$ | 0.63 |
| ZnO | 0.03 | MgO | 0.36 |
| $K_2O$ | 0.01 | ZnO | 0.33 |
| $TiO_2$ | 0.02 | $K_2O$ | 0.23 |
| MgO | 80 PPM | $TiO_2$ | 0.15 |
| Cl | 81 PPM | Cl | 0.15 |
| NiO | 58 PPM | NiO | 0.07 |
| MnO | 28 PPM | MnO | 0.03 |
| $SnO_2$ | 47 PPM | $SnO_2$ | 0.03 |
| $Cr_2O_3$ | 8 PPM | $Cr_2O_3$ | 0.03 |
| $CeO_2$ | 23 PPM | CuO | 0.03 |
| $Bi_2O_3$ | 17 PPM | $CeO_2$ | 0.02 |
| $ZrO_2$ | 12 PPM | $Bi_2O_3$ | 0.02 |
| BaO | 19 PPM | $ZrO_2$ | 0.02 |
| Br | 8 PPM | BaO | 0.02 |
| $MoO_3$ | 3 PPM | $Y_2O_3$ | 87 PPM |
| | | SrO | 73 PPM |
| | | PbO | 59 PPM |
| | | Br | 17 PPM |
| | | $As_2O_3$ | 7 PPM |
| | | $Nb_2O_5$ | 7PPM |
| | | $Rb_2O$ | 5 PPM |

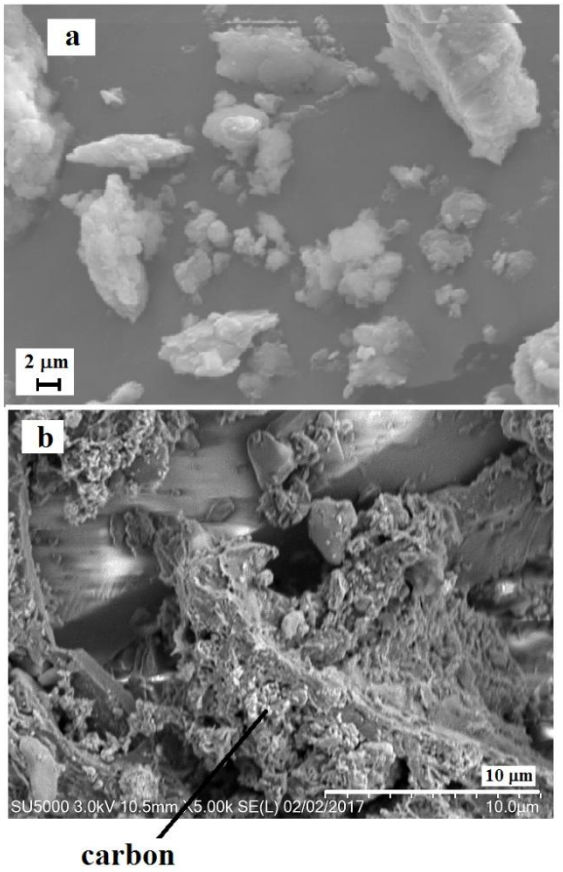


**Fig 2: SEM micrographs of sludge before (a) and after treatment in argon at $T_{tr.}$ = 800°C for 60 min in argon.**

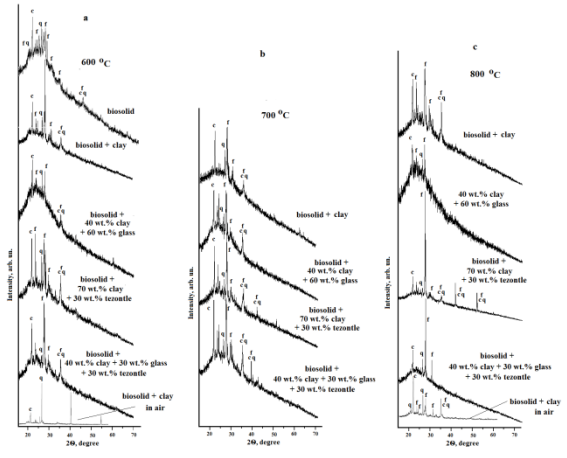


**Fig 3: X-ray diffraction patterns of specimens obtained from different mixtures at different sintering temperatures in an argon atmosphere. The sintering time is 4 h.**

The change in the intensity of the carbon signal (halo) (see Fig. 4) can be explained by the peculiarities of formation of disordered carbon under the conditions of melt appearance and by release of gaseous products of thermal destruction through melt of different viscosity. For instance, for pure clay, an increase in the carbon

content to a treatment temperature ($T_{tr.}$) of 700 ºC (which is characteristic of the thermal destruction process of organic matters [33-35]) was observed (see Fig. 4 a), and at $T_{tr.}$> 700 ºC, the processes of melt formation and carbothermal reduction of oxides, accompanied by gas release, are initiated [35, 36]. In the binary and ternary mixtures, the development of the aforementioned processes depends on the appearance of eutectic melts and their melting points (see Fig. 4 b, c).

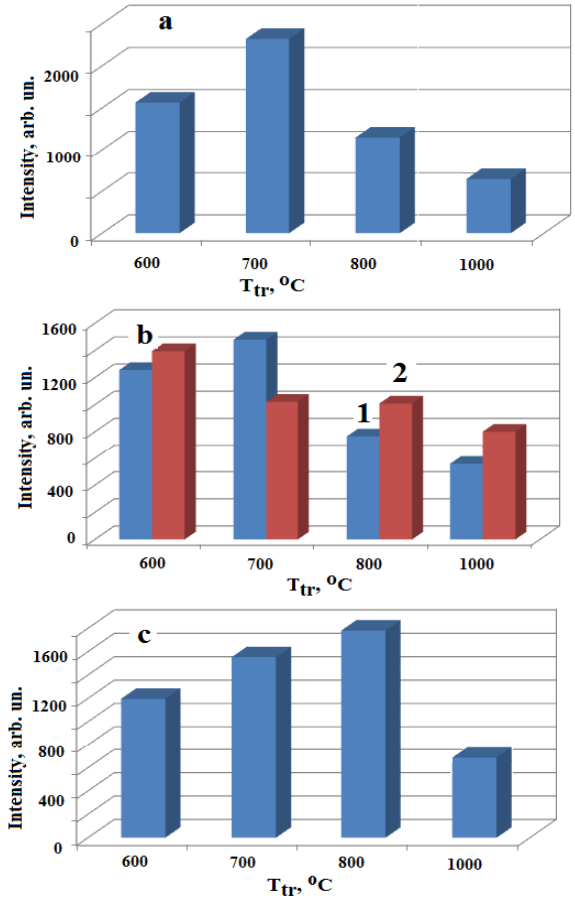


**Fig 4: Change in the intensity of halo from amorphous carbon in clay with the sintering temperature of specimens in an argon atmosphere: (a) red clay + WAS; (b) 40 wt. % red clay + 60 wt. % glass + WAS (1); 70 wt. % red clay + 30 wt. % tezontle + WAS (2); (c) 40 wt.% red clay + 30 wt. % glass + 30 wt. % tezontle + WAS. The sintering time is 4 h.**

SEM studies showed that, independently of the composition of the used mixtures, sintering temperature, and sintering time, all synthesized ceramic specimens have large porosity (Fig. 5).

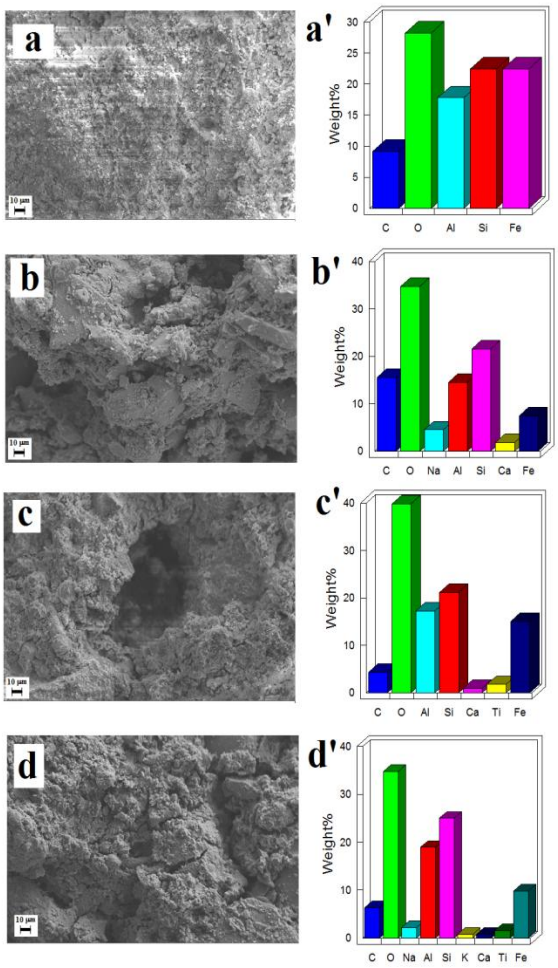


**Fig 5: Micrographs of fractures of specimens (a-d) and contents of elements in the registered region (a'-d'). (a, a') red clay + WAS; (b, b') 40 wt. % red clay + 60 wt. % glass + WAS (1); (c, c') 70 wt. % red clay + 30 wt. % tezontle + WAS; (d, d') 40 wt.% red clay + 30 wt. % glass + 30 wt. % tezontle + WAS. $T_{sint}$ = 1000 ºC, $t_{sint}$ = 8 h.**

The elementary composition of the specimens correlates with that in the used components of mixtures: O, Si, Al, Fe, K, Ca, Na, Mg, and Ti. Moreover, carbon is present in the specimens in different amounts. As is seen in Fig. 6, carbon is distributed over the whole volume of the specimens and on pore boundaries (and, therefore, on the surface of pores). It is worth noting that the carbon content in specimens obtained with the use of glass additives turns to be higher (see Fig. 6, specimens No. 3–5).

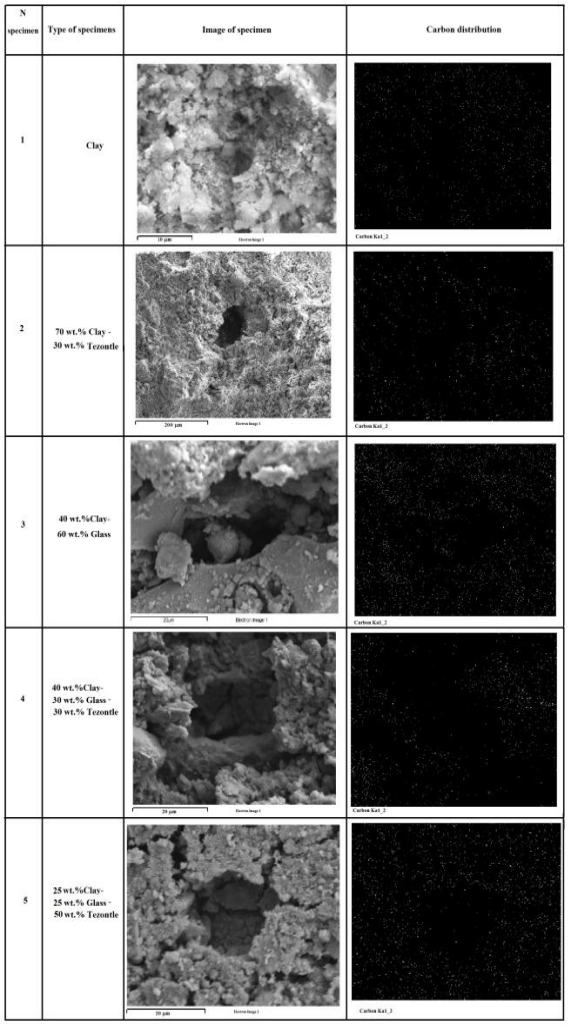


**Fig 6: Micrographs of fractures of specimens and a carbon distribution in the registered region. $T_{sint.}$ = 750 °C, $t_{sint.}$= 9 h.**

This is caused by the fact that, after the appearance of glass melt (or eutectic melt), a part of formed carbon is "encapsulated" (isolated from the external gaseous atmosphere) and turns to be covered by the melt. Such situation is characteristic of relatively low sintering temperatures ($T \leq 800$ ºC) and small temperature treatment times ($t \leq 4$ h) (Fig. 7 b, c). In the case of a shortage of the melt, a substantial fraction of formed carbon is removed from the porous material (Fig. 6, specimens No. 1, 2, and Fig. 7 a). With increase in the sintering temperature ($T \geq 800$ ºC) under a shortage of the melt, the process of carbothermal reduction of oxide phases occurs, which leads to a decrease in the carbon content (see Fig. 7). At the same time, at a large content of eutectic melts, which is typical of ternary mixtures, even at $T_{sint.}$ = 1000 ºC, carbon is retained in the ceramics (Fig. 7 c).

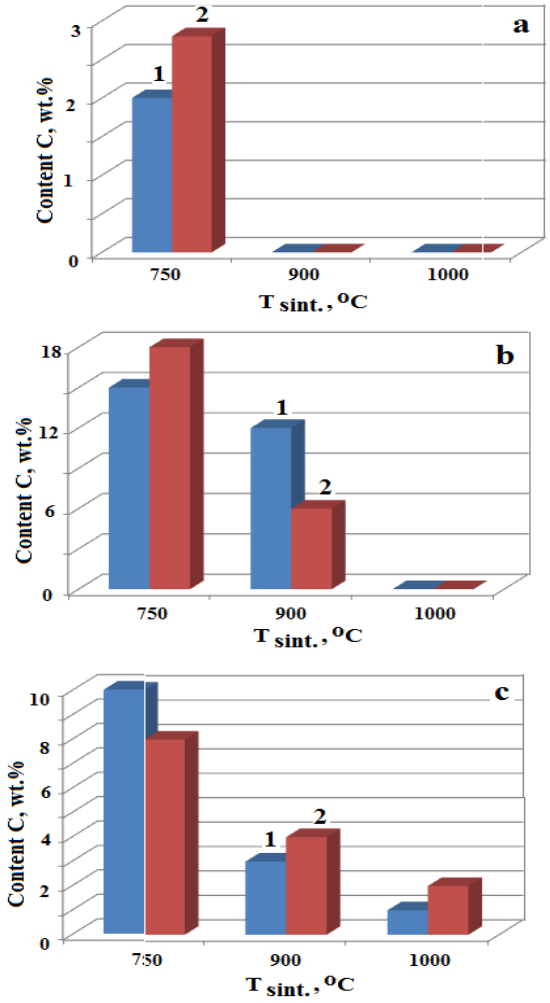


**Fig 7: Carbon content in specimens depending on the sintering temperature. $t_{sint.}$ = 4 h. In (a): (1) for 100 wt. % clay; (2) for 70 wt. % clay–30 wt. % tezontle. In (b): (1) for 50 wt. % clay–50 wt. % glass; (2) for 40 wt. % clay–60 wt. % glass. In (c): (1) for 40 wt. % clay–30 wt. % glass–30 wt. % tezontle; (2) for 25 wt. % clay–25 wt. % glass–50 wt. % tezontle.**

The investigation of water absorption (Figs. 8, 9) also indicates that open porosity is typical of the synthesized specimens. The absorbability depends on the sintering temperature, sintering time, and composition of the specimens. On the whole, it depends on the content of the formed melt during sintering and filling of the pore space by the melt. In other words, with increase in the sintering temperature and temperature treatment time, the amount of the formed melt increases, and its viscosity decreases, thus intensifying the pore healing process.

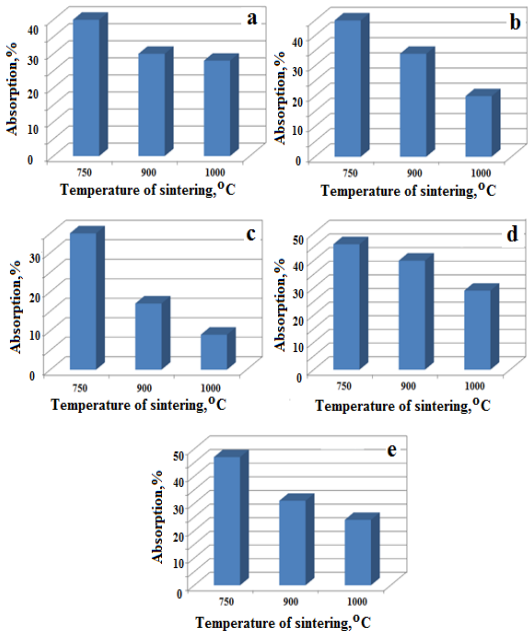


**Fig 8: Change in water absorption with the sintering temperature of specimens of different composition. (a) for 100 wt. % clay; (b) for 70 wt. % clay–30 wt.% tezontle; (c) for 40 wt. % clay–30 wt. % glass–30 wt. % tezontle; (d) 25 wt. % clay–25 wt. % glass–25 wt. % tezontle; (e) for 25 wt. % red clay + 25 wt. % glass + 50 wt. %tezontle. $t_{sint.}$= 4 h.**

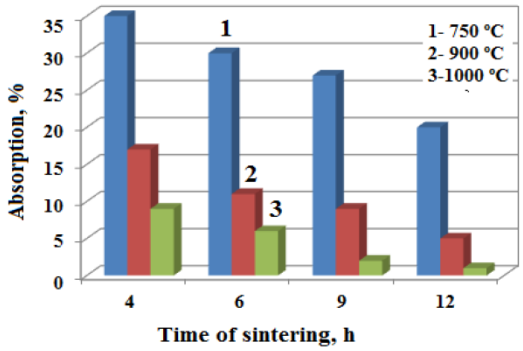


**Fig 9: Water absorption by specimens obtained from the 40 wt. % clay–60 wt. % glass mixture in different sintering regimes: (1) $T_{sint}$= 750 °C, (2) $T_{sint}$= 900 °C, (3) $T_{sint}$= 1000 °C.**

## 3.3 Properties of carbonized materials

### 3.3.1 Ceramics

Performed investigations of the sintering process of red clay–glass–tezontle–WAS composite mixtures in the temperature range 850–1000 °C for 4–12 h showed that a carbonized porous material formed. Though the phase composition of the synthesized ceramics is identical to that of traditional wall ceramics [16-18], it was necessary to determine its strength properties because the sintering temperature and time differ substantially from those in traditional regimes. Moreover, the ceramic is highly porous. Testing results of specimens are presented in Table 2**.** These results indicate that, within the framework of the developed technology, porous ceramics of different application, namely, a wall material, haydite, and a filtering material, can be synthesized.

**Table 2. The properties of specimens obtained with use of WAS**

| Type of initial Mixtu-res, wt. % | T, °C | t, h | Con-tent WAS, g | Compression Kg/cm$^2$ | Flexion Kg/cm$^2$ | W, % | Applica-tion |
|---|---|---|---|---|---|---|---|
| red clay standart | 1000 | 48-70 | 0 | 75-300 | 18-28 | 5- 7 | wall brick |
| red clay | 950<br>1000 | 48<br>48 | 60<br>40 | 27<br>30 | | 25<br>12 | for filtration<br>wall brick |
| 70 red clay + 30 tezontle | 900<br>1000 | 6<br>12<br>8<br>12 | 40<br>40<br>40<br>40 | 40<br>80<br>130.6<br>114.8 | 74.13<br>51.23 | 15<br>5<br>23.6<br>22.24 | for filtration<br>wall brick<br>wall brick<br>-"- |
| 50 red clay + 50 glass | 1000 | 8<br>12 | 40<br>40 | 275.3<br>102.6 | 72.6 | 16.32<br>18.3 | wall brick<br>-"- |
| 40 red clay + 60 glass | 850<br>950<br>1000 | 6<br>8<br>8 | 60<br>60<br>60 | 60<br>60<br>60 | 25<br>60<br>80 | 15<br><1<br><1 | for filtration<br>as clinker brick |
| 40 red clay + 30 glass+ 30 tezontle | 950<br>1000 | 8<br>8<br>12 | 60<br>60<br>40 | 60<br>100<br>81.4 | | 14<br>10<br>21.2 | wall brick<br>-"-<br>-"- |
| 25 red clay + 25 glass + 50 tezontle | 1000 | 8<br>12 | 40<br>40 | 87<br>26 | 44.3<br>114.7 | 25.4<br>24.7 | wall brick |

### 3.3.2 Adsorbing ceramic material

The presence of free carbon in the ceramic material enables us to assume that it can be used not only as heat-insulating filler, but also as an adsorbent. However, such material must contain a substantial amount of carbon and, hence, must be obtained by low-temperature short-term treatment of mixtures.

Performed tests of specimens sintered in the range 600–800 ºC for 1 h showed that they have low strength, high porosity and exhibit high water absorption. This means that these specimens have high open porosity. In the indicated temperature range, the largest amount of the reactive carbon material (product of thermal destruction of WAS) is retained [33]. According to the microanalysis data, its content in different specimens ranges from 5 to 20 wt. %.

Experiments on adsorption of MB showed that this material can absorb the dye from aqueous solution (Figs. 10, 11). A characteristic feature of this type of carbonized specimens is their slow adsorption. The obtained results indicated that the dye is most efficiently absorbed by specimens in which a liquid (glass or eutectic) phase does not form and, hence, carbon encapsulation does not occur in the used sintering regime. It can be suggested that centers of adsorption are carbon located on the surface of pores (see Fig. 6).

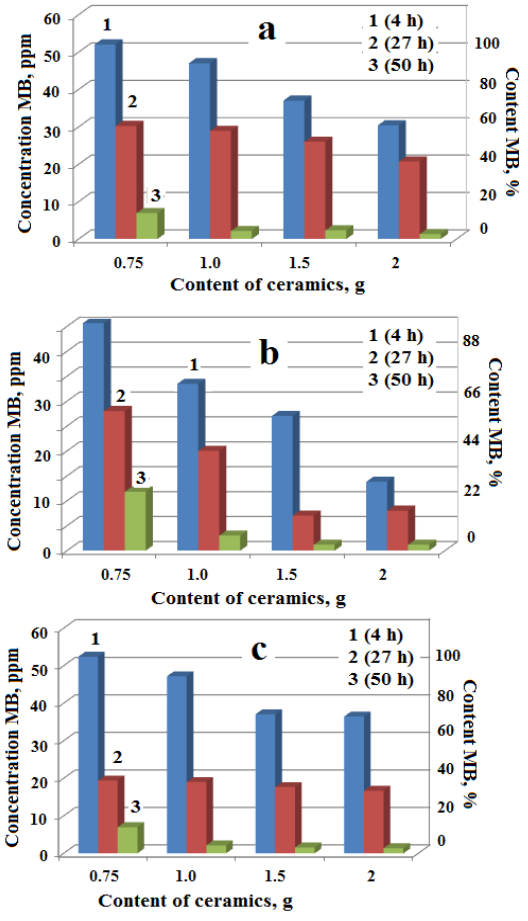


**Fig 10: Change in the content of MB in aqueous solutions depending on the content of ceramics obtained from: (a) 100 wt. % clay; (b) 70 wt. % clay–30 wt. % tezontle mixture; (c) 40 wt.% clay–60 wt.% glass mixture after holding in solution: (1) for 4 h; (2) for 27 h; (3) for 50 h. Specimens obtained at $T_{sint.}$= 700 ºC, $t_{sint.}$ = 1 h. The volume of solution is 20 ml. Initial concentrations MB in solution ($C_{MB}$) 50 ppm.**

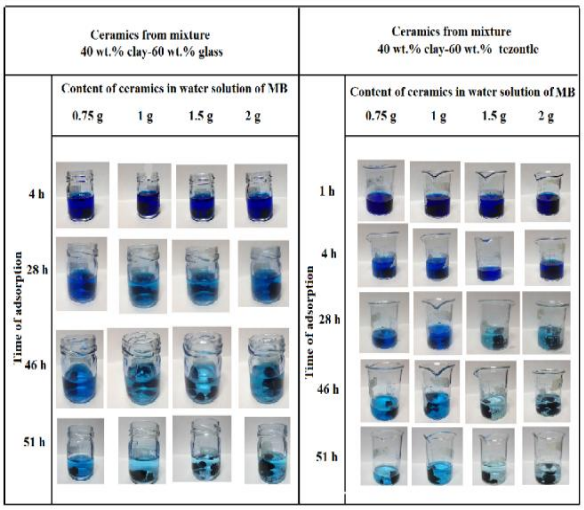


**Fig 11: Change in the color of the aqueous solution due to MB adsorption by carbonized ceramics. Initial concentration of MB 50 PPM/20 ml water. For ceramics: $T_{sint.}$ = 700 ºC, $t_{sint.}$ = 1 h.**

Thus, sintering of the investigated mixtures in an argon atmosphere (and, hence, in vacuum and/or under conditions of limited access for air in burning kilns, makes it possible not only to realize an energy- and water-saving technology of synthesis of ceramic products with different strength properties, but also obtain a new type of low-temperature ceramics with specific adsorption properties, which "free" carbon imparts to it.

## 4. Conclusions

The sintering of clay-WAS and the clay–tezontle–WAS, clay–glass–WAS, and clay–tezontle-glass–WAS mixtures in an inert atmosphere (or in conditions of oxygen deficit) in the temperature range 600–1000 ºC is accompanied by the formation of amorphous carbon in the ceramic material.

The carbon content in specimens, their porosity, and water absorption depend on the composition of the initial mixtures, sintering temperature, and sintering time.

During sintering in the range 750–1000 ºC for decreased sintering time of 6–12 h, brick products with good mechanical properties, which cannot be achieved during sintering in air in this regime, form.

During sintering in the range 600–800 ºC for 1 h, a low-strength porous ceramic product containing a large amount of free carbon forms. This material exhibits adsorption properties and can be used for the purification of industrial sewage water from dyes.

Specimens free from glass additives have the highest adsorption properties, and specimens formed by the mechanism of liquid phase–solid phase sintering and

containing glass and tezontle in initial mixtures exhibit the best strength properties.

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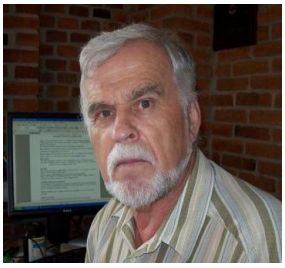

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